ONLINE ISSN 2069-7430
ISSN-L 1841-4737
PRO LIGNO
www.proligno.ro
Vol. 13 N° 4 2017
pp. 115-122

DURABILITY OF THERMO-HYDRO TREATED (THT) BIRCH VENEERS AND PLYWOOD

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Abstract:

The effect of thermo-hydro treatment (THT) on the durability of birch wood veneers and two plywood products – A and B was studied. THT was carried out in a multi-functional pilot scale wood modification device under elevated water vapour pressure at the following temperatures and treatment times (°C/min): 150/10; 150/50; 160/10 and 160/50. After THT, durability was examined according to the standards ENV 12038, EN 84 and EN 73. The durability class (DC) according to the CEN/TS 15083-1 was assigned to each treatment after fungal exposure. Water soluble extractives from THT samples were analysed with a UV-Vis Spectrometer in a range of 200 - 400nm, potentiometric titration and the Malaprade reaction.

The best DC (2 - 3) for aged veneers was reached with the THT 160°C against the white rot fungus. The THT of industrial plywood A at 160°C enabled producing a material with improved durability (DC 1 - 3) after ageing. Plywood B with glued THT veneers showed lower decay resistance than plywood A. After ageing, better performance of plywood B was achieved at the THT 160°C assigning to DC 2 - 4.

At the THT regimes under study, water soluble products (acids, sugars, aromatic compounds) were formed from both polysaccharides and lignin.

Key words: *Thermo-hydro treatment (THT); decay fungi; durability; veneer; plywood.*

INTRODUCTION

Plywood is an engineered wood-based product with improved physical and mechanical properties including high strength-to-weight and strength-to-thickness ratios, and excellent dimensional stability. Plywood panels are of great importance for furniture production and building construction and have been widely used for a variety of interior and exterior applications.

The properties of plywood depend on the quality of the different layers of veneer, the order of layer placement, the adhesive used, and the control of bonding conditions (Youngquist 1999). However, the application of plywood in exterior conditions is limited due to the sensitivity to moisture and biodegradation (Baileys et al. 2003).

Thermo-hydro treatment (THT) of wood at relatively high temperatures ranging from 150°C to 260°C is an alternative and environmentally friendly protection method in comparison to the chemical treatment with biocides. Temperatures around 160°C for hardwoods and around 180°C for softwoods are common (Militz 2002). An important effect achieved by the THT of wood is reduced hygroscopicity, and subsequently, increased dimensional stability (Biziks et al. 2015; Hillis 1984; Zaman et al. 2000) and improved durability (Boonstra et al. 2007; Irbe et al. 2014; Kamdem et al. 2002). Besides, the chemical, structural and mechanical properties of wood are changed (Andersons et al. 2016a; Biziks et al. 2016; Hill 2006).

There is limited information about the impact of THT on wood-based products. Several studies on plywood from thermally modified beech (Grzeskiewicz et al. 2009; Schulz et al. 2012), poplar veneers (Zdravković et al. 2013) and the durability of different commercial wood-based products (Barnes et al. 2016) are reported. However, an industrial method has not yet been developed.

We have examined two approaches for producing THT plywood. The first is the THT of industrial plywood panels and the second is the THT of veneers prior to gluing to form a plywood panel. Thin veneers treated in an aggressive treatment environment (pressure, temperature and low pH values) react differently compared to solid wood. High treatment temperatures and exposure times, which are suitable for solid wood,

are not suitable for veneers, because they become more brittle, resulting in a high loss of mechanical strength. Therefore, mild treatment parameters were chosen for veneers and plywood. Our investigations on different properties of THT veneers and two THT plywood products (A and B) have been carried out and partially published (Andersons et al. 2016b; Grinins et al. 2016a; Grinins et al. 2016b).

OBJECTIVE

In the present study, the durability of THT veneers and two THT plywood products – A and B were investigated according to the standards ENV 12038, EN 84 and EN 73. The thermal destruction products in water leachates were analysed and their effect on decay resistance was discussed.

MATERIAL, METHOD, EQUIPMENT

THT Procedure

THT plywood was obtained by two different methods. Plywood A represented THT industrial birch plywood glued with a phenol formaldehyde (PF) adhesive. Plywood B represented THT birch veneers glued with a PF film (temperature – 140°C, pressing time – 15 min, pressure – 1.2 MPa) to make plywood panels. The parameters of plywood A and B are given in Table 1.

Table 1

Parameters of plywood panels A and B

Plywood	THT applied	Panel dimensions [mm^3]	Plies [No]	Veneer thickness [mm]	Glue	Replicates [No]
A	Industrial panels	350 × 1000 × 18	13	1.4	PF adhesive	6
B	Veneer sheets	350 x 900 x 13	9	1.4	PF film, 220 g/m^2	6

THT was carried out in a pilot scale 540 L autoclave (WTT, Wood Treatment Technology) at elevated water vapour pressure, at four treatment regimes (max temperature, ˚C/ time, min): 150/10; 150/50; 160/10/ 160/50. The mild treatment regimes were chosen taking into account the sensitivity of birch solid wood against THT in our previous experiments, and the small thickness of veneers.

THT of the materials was carried out in three steps: heating, holding at the given temperature, and cooling. A calculated amount of water was pumped in at the beginning to generate saturated steam. Prior to the heating step, samples were held for 30min under a 0.02 MPa pressure in an autoclave for oxygen content reduction. The total time of treatment including the cooling down to room temperature depends on T_{max}, i.e. 24 - 25h (150°C) and 26 - 27h (160°C).

THT veneers and plywood panels were conditioned at a temperature of 20 ± 2°C and a 65 ± 5% relative humidity (RH) to equilibrium moisture content (MC).

Decay Test

The decay resistance was determined for (i) THT veneers with dimensions of 50×25×1.4mm^3, and (ii) THT plywood A and B with dimensions of 50×25×thickness (mm^3) according to the modified European Prestandard ENV 12038:2002. Six specimens for each treatment were exposed to the brown rot fungus *Coniophora puteana* (BAM Ebw 15) and the white rot fungus *Trametes versicolor* (CTB 836A). Pine and birch wood specimens were used as virulence controls for *C. puteana* and *T. versicolor,* respectively. The fungi were cultivated on a medium containing 5% malt extract concentrate and 2% Fluka agar. In the THT veneer test, sterilised specimens were aseptically placed on 3mm steel supports in Petri dishes, and incubated at 22 ± 2°C and 70 ± 5% RH for 6 weeks. In the THT plywood test, the sterile specimens were placed on glass supports in Kolle flasks and cultivated for 16 weeks.

Subsequent to cultivation, the specimens were removed from the culture vessels, brushed free of mycelium and oven dried at 103±2°C. The percentage mass loss (ML) of the specimens was the measure for the extent of fungal degradation.

Durability class (DC) according to the Technical Specification CEN/TS 15083-1:2005 was assigned to each treatment after fungal exposure: 1 – very durable (ML ≤ 5%); 2 – durable (ML > 5 to ≤ 10%); 3 – moderately durable (ML > 10 to ≤ 15%); 4 – slightly durable (ML > 15 to ≤ 30%); 5 – not durable (ML > 30%).

Ageing Procedures: Leaching and Evaporation

Prior to the ENV 12038 test, six THT veneers and plywood specimens from each treatment were leached with distilled water according to EN 84:1997. Additionally, the water extractive of the polymerised PF adhesive was obtained. UV absorption of diluted extracts was measured in a range of 200 - 400nm with UV-VIS Spectrometer Genesys™ 10. Acetic acid was potentiometrically titrated with 0.1M KOH solution using TIM 980 Titration Manager. The α-diol groups for sugar content analyses were determined *via* the reaction with periodate (Malaprade reaction) according to Meile et al. (2014).

Before the ENV 12038 test, six THT plywood specimens from each treatment were evaporated for 12 weeks in a wind channel according to EN 73:1988.

RESULTS AND DISCUSSION

Decay Resistance of THT Veneers

Decay test results for veneers are shown in Table 2. The ML of veneers (in both test procedures) is higher when subjected to *C. puteana;* possibly, because the virulence control shows a 43.4% ML vs. a 25.0% ML for *T. versicolor*. ENV 12038 results show that the lowest decay resistance against *C. puteana* is exhibited for veneers 150/10 (43%), whereas in the case of *T. versicolor,* the untreated and both 150/10 and 150/50 samples show a ML of 29%. T_{max} 160°C is most effective with this regard. After attack by *C. puteana,* the average ML of these veneers is 30%, but for *T. versicolor* 19%.

Table 2

Mass loss with SD and durability class (DC) of THT birch veneers after degradation by the decay fungi C. puteana and T. versicolor according to the test methods ENV 12038 and EN 84. Mean values, n = 6

Treatment temp./ time [°C/ min]	ENV 12038		ENV 12038 with EN 84	
	C. puteana	*T. versicolor*	*C. puteana*	*T. versicolor*
Untreated	36.6 ± 3.5 (DC 5)	29.6 ± 3.4 (DC 4)	30.6 ± 5.6 (DC 5)	9.7 ± 3.4 (DC 2)
150/10	43.0 ± 6.8 (DC 5)	29.3 ± 4.2 (DC 4)	31.3 ± 8.6 (DC 5)	18.4 ± 6.0 (DC 4)
150/50	38.6 ± 4.9 (DC 5)	28.8 ± 8.0 (DC 4)	40.7 ± 6.6 (DC 5)	18.8 ± 4.9 (DC 4)
160/10	29.1 ± 4.4 (DC 4)	19.6 ± 5.0 (DC 4)	27.9 ± 2.2 (DC 4)	12.1 ± 3.7 (DC 3)
160/50	30.7 ± 12.5 (DC 5)	18.1 ± 5.5 (DC 4)	18.8 ± 6.6 (DC 4)	6.7 ± 3.0 (DC 2)

In the combined ENV 12038/ EN 84 test, veneers 150/50 show the lowest decay resistance against *C. puteana* (40.7%), while both veneers 150/10 and 150/50 have the lowest decay resistance against *T. versicolor* (18%). Expectedly, the best ML results are obtained for veneers 160/50, namely 18.8% (*C. puteana*) and 6.7% (*T. versicolor*).

According to the CEN/TS 15083-1, the veneers treated at the given regimes are slightly durable to non-durable (DC 4 - 5) after the ENV 12038 test, and durable to non-durable (DC 2 - 5) after the leaching test (Table 2).

Our previous experiments with THT birch solid wood, treated at 160/3h and 170/1h, resulted in more durable samples against decay fungi (Irbe et al. 2014). The lower thickness (1.4mm) of the veneers tested in the present paper is one of the reasons for the differences, compared with the case of the experiments with solid wood samples (5mm thickness). Another reason could be the milder THT regimes tested in this study.

After leaching, water extractives were collected and analysed by UV-Vis spectrometry (Fig. 1): one band was around 202 - 206nm and another around 276 - 280nm. These bands are characteristic for aromatic compounds, and can be connected with the leaching of soluble lignin derivatives, polyphenol type compounds and/or furans, i.e. the destruction products of wood components (lignin, hemicelluloses). The extractives obtained from the veneers 160/50 have the highest absorptions, indicating that water soluble thermal degradation products are formed. The samples 160/10 have a lower absorbance but both samples 150/10 and 150/50 have the lowest values. Accordingly, serious degradation begins at 160°C. Extractives from the untreated sample do not display a band at 280nm and have a very low absorbance at 204nm.

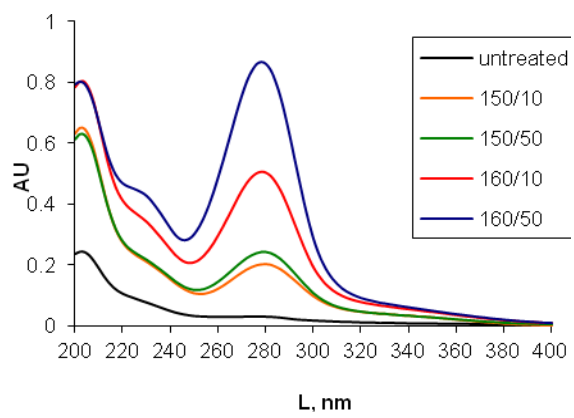

Fig. 1.
UV-Vis spectra of water extractives obtained from THT veneers.

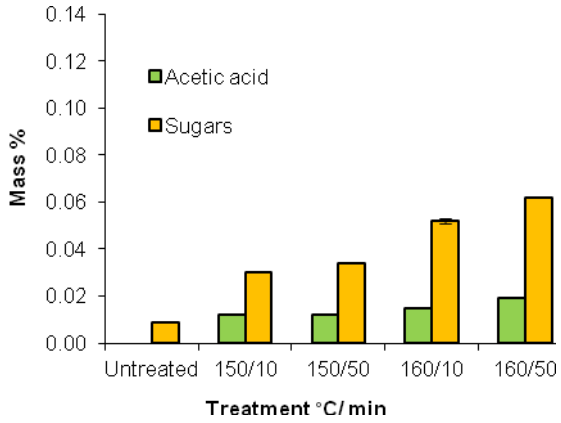

Fig. 2.
Total sugar (expressed as hexoses and pentoses) and acetic acid concentration in water extractives of veneers.

The origin of the UV bands is not always unambiguous as furfural and hydroxymethyl furfural and their condensation products are UV active, mainly at 280nm (Hon and Shiraishi 2000). Therefore, the more typical lignin band at 205nm is a better choice for lignin estimation, and this peak clearly shows that the THT time has no effect on lignin splitting, while a higher temperature increases the intensity of the 205nm peak.

Fig. 2 also demonstrates the efficiency increment from 150 to 160°C. However, the total amounts of water solubles are very small. These products are generated after hemicelluloses destruction – acetic acid is formed from acetyl groups, which split off during the thermal treatment and work as a catalyst for further depolymerisation of polysaccharides, generating monosaccharides (sugars) (Tjeerdsma et al. 1998). Our results partly explain the better decay resistance of veneers after leaching according to EN 84, i.e. the easily accessible products for enzymatic activity are removed by leaching.

The thermal degradation products are not toxic for both fungi as the ML after veneer leaching does not change much in the case of *C. puteana,* but for *T. versicolor,* ML even decreases probably because of leached nutrition compounds such as sugars etc. Untreated veneers demonstrate a similar trend, with a decreased decay rate after leaching.

Decay Resistance of THT Plywood A

The ML of THT plywood A in the test ENV 12038, and in the combined ENV 12038/ EN 73 test is higher when subjected to *T. versicolor*, but for the combined ENV 12038/ EN 84 test, it is higher for *C. puteana* (Table 3). In ENV 12038 and combined 12038/ EN 73 tests, both untreated and THT plywood A is very durable to durable (DC 1 - 2) to attack by fungi.

In the combined ENV 12038/ EN 84 procedure, the untreated plywood shows the lowest decay resistance against both fungi. The THT 150°C provides a low resistance against *C. puteana* (ML 26.3 - 31.7%) and *T. versicolor* (ML 18.0 - 21.3%). Better resistance against both fungi is achieved at the THT 160°C (DC 2 - 3). Despite this improvement, plywood A cannot be considered as very durable because the ML of the specimens is greater than 5% (8.4 - 14.5%).

Two bands from the UV spectra at 202 - 206nm and 276 - 280nm were analysed (Fig. 3). Extractives from the THT 160°C have the highest absorption for both bands, indicating that water soluble thermal destruction products are formed. Acid catalysed degradation of hemicelluloses leads to the formation of formaldehyde, furfurals and other aldehydes (Tjeerdsma et al. 1998). These hemicellulose destruction products more reliably show an absorption maximum at 280nm but the changes in the lignin structure are represented by the peak at 202 - 206nm. Although lignin is considered as the most thermally stable component of wood, its structure is changed by THT (Grinins et al. 2013).

Table 3

Mass loss with SD and durability class (DC) of THT plywood A after degradation by the decay fungi C. puteana and T. versicolor according to the test methods ENV 12038, EN 84 and EN 73. Mean values, n = 6

Treatment temp./ time [°C/ min]	ENV 12038		ENV 12038 with EN 84		ENV 12038 with EN 73	
	C. puteana	*T. versicolor*	*C. puteana*	*T. versicolor*	*C. puteana*	*T. versicolor*
Untreated	0.9 ± 0.3 (DC 1)	1.1 ± 0.3 (DC 1)	43.5 ± 1.6 (DC 5)	26.7 ± 1.1 (DC 4)	3.4 ± 0.4 (DC 1)	3.6 ± 0.2 (DC 1)
150/10	1.8 ± 0.7 (DC 1)	4.5 ± 3.9 (DC 1)	31.7 ± 4.8 (DC 5)	21.3 ± 1.5 (DC 4)	1.6 ± 0.3 (DC 1)	5.6 ± 7.2 (DC 2)
150/50	2.9 ± 0.8 (DC 1)	5.4 ± 3.7 (DC 2)	26.3 ± 5.5 (DC 4)	18.0 ± 2.0 (DC 4)	1.1 ± 0.2 (DC 1)	3.6 ± 0.5 (DC 1)
160/10	2.3 ± 0.3 (DC 1)	3.0 ± 1.1 (DC 1)	14.5 ± 2.6 (DC 3)	8.4 ± 0.8 (DC 2)	0.3 ± 0.3 (DC 1)	2.7 ± 0.2 (DC 1)
160/50	2.4 ± 0.6 (DC 1)	2.7 ± 1.1 (DC 1)	10.2 ± 4.7 (DC 3)	9.0 ± 1.0 (DC 2)	0.0 ± 0.3 (DC 1)	1.3 ± 0.1 (DC 1)

Untreated plywood shows no absorption at 280nm and low absorption at 204nm if compared with the THT sample peaks. Separate analyses of the water leachate of the polymerised PF adhesive demonstrated a higher UV absorption at 204nm than at 286nm (data not shown). Accordingly, the effect of the PF adhesive on the UV absorption spectra at 204nm cannot be excluded.

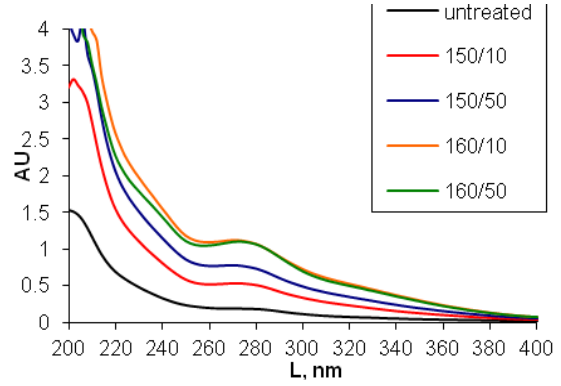

Fig. 3.
UV-Vis spectra of water extractives obtained from plywood A.

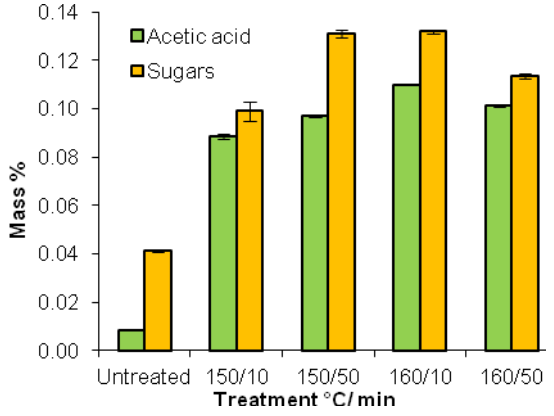

Fig. 4.
Total sugar (hexoses and pentoses) and acetic acid concentration in water extractives of plywood A.

The analyses of water extractives show the presence of some thermal destruction products (sugars and acid), with a tendency to increase with increasing THT regime (Fig. 4). These products are generated after hemicellulose destruction as described before. In plywood A, extracted sugars and acid amounts are higher than for THT veneers. At 160/50, the extracted amount slightly decreases, probably, because the THT time was long enough and decomposition continued, generating simple products like water and carbon dioxide or other easily volatile destruction products.

A significantly higher durability is observed for non-leached THT plywood A if compared with THT non-leached veneers (Table 2). Consequently, the fungal growth perhaps is influenced by the fungicidal effect of the PF adhesive (phenols, formaldehyde) rather than the THT destruction products. After leaching, the water soluble phenolic compounds from the adhesive are removed and plywood becomes susceptible to fungal attack similar to THT veneers.

Decay Resistance of THT Plywood B

The decay resistance of THT plywood B is similar to that of THT veneers (Table 4). The durability of plywood B is much lower than for plywood A after ENV 12038 and combined ENV 12038/ EN 73 tests.

The ML of THT plywood B after ENV 12038 ranges from 13.4% to 33.7% giving DC 3 - 5. In combined 12038/ EN 73, plywood B performs as a moderately durable to slightly durable (DC 3 - 4) material. In the combined ENV 12038/ EN 84, the THT 150°C ensures insufficient resistance against *C. puteana* (ML 29.2 - 32.8%) and *T. versicolor* (ML 17.9 - 27.4%). The best resistance against *T. versicolor* is achieved at the THT 160°C (DC 2 - 3). Nevertheless, plywood B cannot be considered as very durable because the ML is higher than 5% (7.9 - 11.3%). The low durability of plywood B could be attributed to the used PF film, which is made of PF resin impregnated paper. Apparently, the PF film is a more easily accessible substrate for fungal degradation than the PF adhesive in plywood A.

Table 4

Mass loss with SD and durability class (DC) of THT plywood B after degradation by the decay fungi C. puteana and T. versicolor according to the test methods ENV 12038, EN 84 and EN 73. Mean values, n = 6

Treatment temp./ time [°C/ min]	ENV 12038		ENV 12038 with EN 84		ENV 12038 with EN 73	
	C. puteana	*T. versicolor*	*C. puteana*	*T. versicolor*	*C. puteana*	*T. versicolor*
150/10	33.7 ± 4.8 (DC 5)	32.4 ± 2.2 (DC 5)	32.8 ± 2.0 (DC 5)	27.4 ± 4.7 (DC 4)	27.5 ± 6.0 (DC 4)	29.8 ± 3.9 (DC 4)
150/50	32.9 ± 5.0 (DC 5)	28.9 ± 1.4 (DC 4)	29.2 ± 2.2 (DC 4)	17.9 ± 5.9 (DC 4)	27.7 ± 5.6 (DC 4)	28.0 ± 2.7 (DC 4)
160/10	17.0 ± 3.6 (DC 4)	24.3 ± 1.8 (DC 4)	22.0 ± 1.4 (DC 4)	11.3 ± 1.2 (DC 3)	12.6 ± 4.9 (DC 3)	24.4 ± 1.2 (DC 4)
160/50	13.4 ± 1.8 (DC 3)	17.5 ± 1.3 (DC 4)	17.7 ± 3.5 (DC 4)	7.9 ± 1.6 (DC 2)	13.1 ± 5.8 (DC 3)	20.6 ± 2.3 (DC 4)

Similar to the THT veneers and plywood A, the UV bands at 202 - 206nm and 276 - 280nm of plywood B demonstrate the highest absorption for THT 160°C extractives, indicating that water soluble thermal destruction products are formed (Fig. 5). The THT 150°C has lower values.

The analyses of water extractives show the presence of thermal destruction products (sugars and acid), with a tendency to increase with increasing THT regime (Fig. 6). A pronounced amount of sugars is detected in leachates of THT 160°C samples. The mass percentage is three times higher than for THT 150°C samples and twice higher than at an equal regime for THT veneers. Perhaps, the gluing procedure of veneers into the plywood B plates at high temperature (140°C) and pressure (1.2MPa) promotes some decomposition of structural oligomers to monomers.

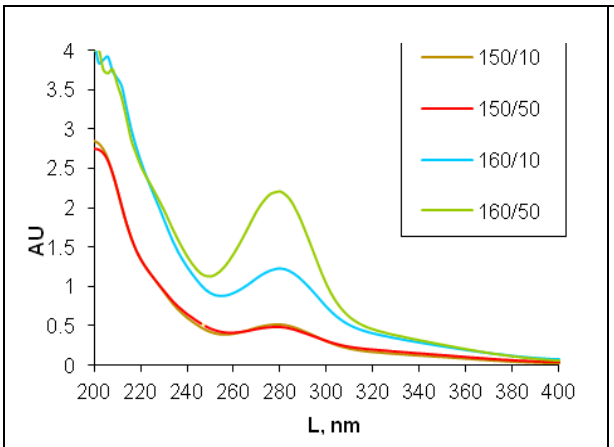

Fig. 5.
UV-VIS spectra of water extractives obtained from plywood B.

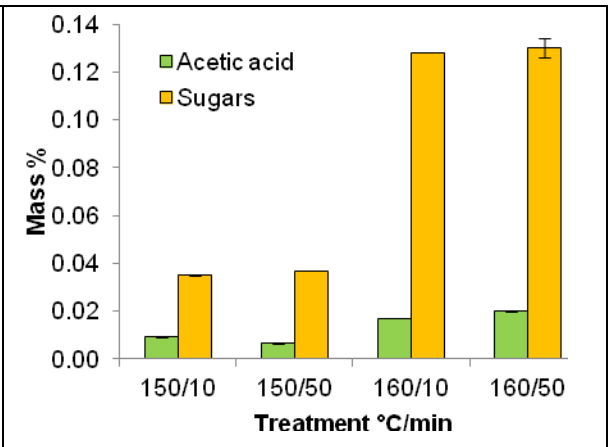

Fig. 6.
Total sugar (hexoses and pentoses) and acetic acid concentration in water extractives of plywood B.

The difference in durability rating between non-leached and leached THT plywood B (Table 4) is not as drastic as for plywood A (Table 3). This can be caused by the non-toxic behaviour of the PF film in comparison with the PF adhesive. Similar to THT veneers, the thermal destruction products have promoted the fungal degradation of plywood B samples (ENV 12038), while after the leaching, the decay capacity dropped.

CONCLUSIONS

The THT of birch veneers in comparatively mild conditions (150 and 160°C; 10 and 50min) does not allow obtaining of a very durable material (ML ≤ 5% according to CEN/TS 15083-1) against wood decay fungi. The best durability class (DC 2 - 3) of veneers is achieved by the THT 160°C against the white rot fungus *T. versicolor*. Water soluble compounds, produced at the given THT regimes, favour the decay ability of white and brown rot fungi because the ML declines after leaching.

The THT of birch plywood A at 160°C enables producing a material with improved resistance to decay fungi in outdoor exposure (moisture and evaporation) that is attributed to DC 1 - 3 (very durable to moderately durable). THT birch plywood B shows a lower decay resistance than plywood A. After the ageing procedures, the best performance of plywood B is achieved at the THT 160°C, attributed to DC 2 - 4 (durable to slightly durable). If compare two approaches of THT plywood producing, it is clear that the THT of veneers prior to the gluing with a PF film makes a product with a lower durability than the THT of industrial plywood which is glued with a PF adhesive.

At the THT regimes under study, substantial component changes occur in birch veneers and plywood, namely, water soluble extractives (acids, sugars, aromatic compounds) are formed from both polysaccharides and lignin.

Untreated birch plywood is very durable (DC 1) before leaching, probably because of the effect of the PF adhesive. After the non-polymerising adhesive components are leached out, the plywood becomes more accessible to fungal attack.

ACKNOWLEDGEMENT

Financial support of the ERAF project “Plywood from thermally modified veneer with improved durability properties” No. 2014/0018/ 2DP/2.1.1.1.0/14/APIA/VIAA/040 and the National Research Programme (ResProd) Project No. 3 “Biomaterials and products from forest resources with versatile applicability” is gratefully acknowledged. The authors thank MSc Kristine Meile (LS Institute of Wood Chemistry) for help in water extractives analyses.

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